

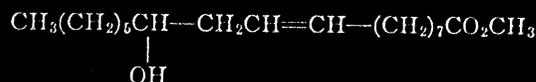
650

responding hydroxamic acids, fractionating these, and reconverting the pure oleohydroxamic acid to oleic acid.

The present procedure<sup>9</sup> is a simplification of the methods of Brown and Shinowara<sup>5</sup> and of Wheeler and Riemenschneider.<sup>6</sup> It gives higher yields of a somewhat lower-quality product.

The procedure described is more satisfactory than those requiring the crystallization of soaps of oleic acid. For a review of these methods see ref. 5.

### METHYL RICINOLEATE

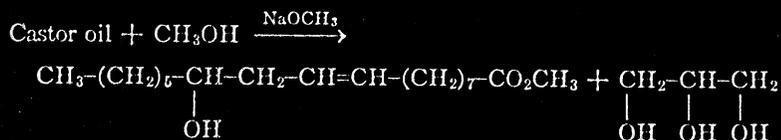


Mol. wt. 312.5 (C<sub>19</sub>H<sub>36</sub>O<sub>3</sub>)

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#### I. Principle



#### II. Procedure

One thousand grams of U.S.P. castor oil are added to a solution of 3 gm. of metallic sodium in 2 l. of anhydrous methanol in a 12-l. Pyrex round-bottomed flask. The reaction mixture is refluxed for 45 minutes and then diluted, with stirring, with about 7 l. of water containing 25 ml. of 6 N hydrochloric acid. The lower aqueous layer is siphoned off and discarded. The methyl esters are transferred to a 4-l. separatory funnel and washed with water until free of chloride ion. The crude, washed esters

<sup>9</sup> D. Swern, H. B. Knight, and T. W. Findley, *Oil & Soap*, **21**, 133 (1944).

are dried by heating to 100-105° under moderate vacuum in a stream of inert gas. The yield at this point is 970-980 gm. of a pale yellow oil. This is fractionally distilled *in vacuo*.<sup>1</sup> The fraction distilling at 185-187°/2 mm. consists of essentially pure methyl ricinoleate. The yield is 670-680 gm. (84-85% of the theory based on the assumption that castor oil contains 80% of ricinoleic acid).

### III. Properties and Purity of Product

Methyl ricinoleate is a colorless, odorless oil melting at -6°. It gives the following constants: refractive index,  $n_D^{20} = 1.463$ ; specific rotation,  $[\alpha]_D^{25} = +5.05^\circ$  (5% solution in acetone); iodine number, 81-82; saponification equivalent, 312-313; hydroxyl, 5.5%.

### IV. Methods of Preparation

Methyl ricinoleate was prepared by methanolysis of castor oil followed by fractional distillation and low-temperature solvent crystallization.<sup>2</sup> The present procedure is that of Kass and Radlove.<sup>3</sup>

<sup>1</sup> A column with a low pressure-drop should be used to avoid pot temperatures much in excess of 220°. In addition, the column should have a fairly high throughput to keep the distillation time as short as possible. A Vigreux column, 3 ft. long and 1 in. in diameter (five theoretical plates), electrically heated and well insulated, is satisfactory. The checkers found that a vacuum-jacketed silvered Vigreux column gave lower yields than an electrically heated one.

<sup>2</sup> J. B. Brown and N. D. Green, *J. Am. Chem. Soc.*, **62**, 738 (1940).

<sup>3</sup> J. P. Kass and S. B. Radlove, *J. Am. Chem. Soc.*, **64**, 2253 (1942).